

PATENT ABSTRACTS OF JAPAN

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(54) PRODUCTION OF IMIDIZED COPOLYMER

(57)Abstract:

PURPOSE: To produce an imidized copolymer having a high impact resistance without decreasing the mol.wt. in the imidizing step by mixing a precursor of the copolymer with an imidizing agent under a specified shear rate in addition to the conventional imidizing conditions.

CONSTITUTION: An imidized copolymer is produced in two steps: the first step wherein a precursor obtd. from maleic anhydride, a vinylarom. compd., and, if necessary, another ethylenically unsatd. compd. is imidized by mixing it with an imidizing agent in a mixing zone under a pressure of 10kg/cm² or higher, at 200-270°C, and with a shear rate of 10-150sec⁻¹ for 2-5min; and the second step wherein the resulting product is transported from the mixing zone to an adjacent removal zone, where low-mol.-wt. impurities are removed under a reduced pressure.

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CLAIMS

[Claim(s)]

[Claim 1] The manufacturing method of an imide-ized copolymer characterized by carrying out the first following process and the second following process.

The first process: Set the imide-ized precursor copolymer which consists of other ethylene nature unsaturated compounds a maleic anhydride, a vinyl aromatic compound, and if needed to a kneading band, and it is 10 kg/cm². Process which kneads with an imide-ized agent under the above pressure, the temperature of 200-270 degrees C, the shear rate of 10-250 sec⁻¹, and the conditions for 2 - 5 minutes, and imide-izes an imide-ized precursor copolymer.

The second process: The process which feeds with the first process product the removal band which is open for free passage to a kneading band, and removes a low-molecular contaminant under reduced pressure.

[Claim 2] The manufacturing method of an imide-ized copolymer according to claim 1 which inserts an imide-ized precursor copolymer in the insertion band which is open for free passage to a kneading band continuously or intermittently, feeds a kneading band with this continuously or intermittently from this insertion band, and is kneaded with an imide-ized agent.

[Claim 3] The manufacturing method of an imide-ized copolymer according to claim 1 or 2 which performs kneading in a kneading band with the said direction rotation completeness engagement mold twin screw extruder.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001] [Background of the Invention]

[Industrial Application] This invention relates to the approach of manufacturing the

imide-ized copolymer which serves as maleimide or N-permutation maleimide, and a vinyl aromatic compound from one or more sorts of other unsaturated compounds according to a request.

[0002]

[Description of the Prior Art] The copolymer which consists of a maleic anhydride, a vinyl aromatic compound, and other unsaturated compounds, especially a maleic-anhydride-styrene acrylonitrile copolymer (SMA resin) have high heat deflection temperature, and show good thermal resistance, and SMA resin has a styrene acrylonitrile copolymer (AS resin), a styrene-acrylonitrile rubber elastic body graft copolymer (ABS plastics), etc. and the outstanding compatibility. therefore, SMA resin is blended with an AS resin, ABS plastics, etc., and it is used in order to improve thermal resistance, solvent resistance, etc. of an AS resin etc. however, the blend resin with SMA resin, an AS resin, ABS plastics, etc. is difficult for using as an injection-molding ingredient with an elevated temperature and the need of heating coloring and foaming at 230 degrees C or more on the occasion of shaping since a lifting, weight reduction, crosslinking reaction, etc. are shown if it heats especially at 230 degrees C or more. On the other hand, this invention persons have already proposed the approach of manufacturing continuously economically the imide-ized copolymer of the amount of macromolecules which has thermal resistance and high temperature oxidation stability-proof by the single step of a non-drainage system, as a means to solve an above-mentioned trouble, by carrying out predetermined time kneading with an imide-ized agent, and imide-izing a raw material copolymer under a high pressure, (JP,2-4806,A).

[0003]

[Problem(s) to be Solved by the Invention] However, by this invention persons' above-mentioned proposal, the fall prevention effectiveness of the molecular weight of the copolymer in an imide-ized reaction process was not sometimes necessarily enough, and also when the imide-ized copolymer of the amount of giant molecules was obtained even if, there was a case where the shock resistance of blend resin, such as it and ABS plastics, was not attained enough. The place which this invention is made based on an above-mentioned background, and is made into the purpose In the approach of manufacturing continuously economically the imide-ized copolymer of the amount of macromolecules which has thermal resistance and high temperature oxidation stability-proof by the single step of a non-drainage system Without reducing molecular weight substantially at an imide-ized reaction process, as the manufactured imide-ized copolymer has shock resistance high as blend resin with ABS plastics etc., it is offering the approach of imide-izing.

[Summary of the Invention]

[0004]

[Means for Solving the Problem] As a result of doing various test researches that an above-mentioned technical problem should be solved, when carrying out this invention persons giving the shear rate beyond 10 sec-1 for kneading with a raw material copolymer and an imide-ized agent in addition to conditions given in the above-mentioned proposal (JP,2-4806,A), they acquire the knowledge of being effective in the purpose achievement of this invention, and came to complete this invention.

[0005] The manufacturing method of <a summary, i.e., the imide-ized copolymer by this invention,> is characterized by carrying out the first following process and the second following process.

The first process: Set the imide-ized precursor copolymer which consists of other ethylene nature unsaturated compounds a maleic anhydride, a vinyl aromatic compound, and if needed to a kneading band, and it is 10 kg/cm². Process which kneads with an imide-ized agent under the above pressure, the temperature of 200-270 degrees C, the shear rate of 10-250 sec⁻¹, and the conditions for 2 - 5 minutes, and imide-izes an imide-ized precursor copolymer.

The second process: The process which feeds with the first process product the removal band which is open for free passage to a kneading band, and removes low-molecular ***** under reduced pressure.

[0006] According to <effectiveness> this invention, the rate of imide-izing can manufacture continuously economically the imide-ized copolymer of the amount of macromolecules which has thermal resistance and high temperature oxidation stability-proof in the state of [high] homogeneity by the single step of a non-drainage system.

[0007] [Concrete explanation of invention]

the precursor of the imide-ized copolymer which should be carried out [imide]-izing by the approach by <imide-ized precursor copolymer> this invention -- a maleic anhydride, a vinyl aromatic compound, and the need -- responding -- other ethylene nature unsaturated compounds -- since -- it is the becoming copolymer. That is, the precursor copolymer which should be carried out [imide]-izing by this invention is a copolymer of a maleic anhydride, therefore "imide" is maleimide or N-permutation maleimide.

[0008] The comonomer to the maleic anhydride of this precursor copolymer is a vinyl aromatic compound. as a vinyl aromatic compound, a nucleus and (or) a side-chain permutation, or unsubstituted styrene is typical. The substituent in this case has a low-grade alkyl group and halogen atom, a lower alkoxy group, and others. Styrene, alpha methyl styrene, vinyltoluene, ethyl styrene, iso PUROPIRE styrene, and such mixture are in such a vinyl aromatic compound. In this invention, a desirable vinyl aromatic compound is styrene. In addition to the above-mentioned 2 indispensable monomer, this precursor copolymer may copolymerize these and a copolymerizable ethylenic unsaturated monomer further. As such an ethylenic unsaturated monomer, there are things other than the two above-mentioned sorts, for example, (meta), acrylic ester, acrylonitrile (meta), vinyl ester, halogenation vinyl, and others.

[0009] The imide-ized precursor copolymer which should be carried out [imide]-izing in this invention has the desirable thing of the range whose anhydrous maleate content is 10 % of the weight - 40 % of the weight. At less than 10 % of the weight, cannot desire a heat-resistant high thing, and since less than 10% of the weight of a raw material copolymer does not have bad high temperature oxidation stability If it is because there is little semantics which itdares imide-ize and exceeds another side and 40 % of the weight Heating melting of an imide-ized precursor copolymer takes remarkable high temperature (depending on the case, it becomes difficult in a 200 degrees C - 270 degrees C temperature requirement), and the good imide ghost of a

color tone is not obtained, and also it is because imide-ized effectiveness also falls. a vinyl aromatic compound content -- 90 - 60 % of the weight -- desirable -- 90 - 70 % of the weight, and ** -- it is common. the time of copolymerization of it being carried out, as for the ethylenic unsaturated monomer used if needed -- the content - - 1 - 30 % of the weight -- desirable -- 3 - 25 % of the weight, and ** -- it is common. A solution, suspension, and **** can be manufactured with an emulsion-polymerization method as decompose (a pyrolysis or redox decomposition) and, as for this copolymer, a suitable polymerization initiation means, for example, a radical polymerization initiator, should boil all or a part about each of each monomer.

[0010] The "imide-ized agent" as used in the field of <imide-ized agent> this invention is a compound which acts on the acid-anhydride residue of a maleic anhydride, and changes this into imide association, and this is ammonia, the first class, or a secondary amine. The substituent which forms the first class or a secondary amine has the derivative of these hydrocarbons residue that has the substituent which does not have trouble in an aliphatic series (alicyclic group is included), aromatic hydrocarbon residue, or imide-ized reaction. as aliphatic hydrocarbon residue -- about one to ten carbon number -- desirable -- about three to eight thing -- usually -- coming out -- it is -- as aromatic hydrocarbon residue -- carbon numbers 6-12 -- as the substituent to which six to about 8 and ** usually come out, there are, and they do not exert trouble on an imide-ized reaction preferably especially -- a halogen atom, a nitro group, and an alkoxy group -- there are a lower alkoxy group and others preferably.

[0011] If the example of a suitable imide-ized agent to use it by this invention is given, (b) ammonia, (b) fatty amine, for example, monomethylamine, ethylamine, n propylamine, Isopropylamine, a butylamine, an isobutyl amine, sec butylamine, 1, 2-dimethyl propylamine, hexylamine, cyclohexylamine, Aromatic amines (Ha), for example, an aniline, such as an octyl amine and a stearyl amine There are an alt.toluidine, 2 and 4, 6-TORIKURORO aniline, a 4-nitro-2-toluidine, alpha-naphthylamine, beta-naphthylamine, 2-methoxyaniline, 4-methoxyaniline, a 2-ethoxy aniline, a 4-ethoxy aniline, etc. these can be used together by the inside of each group, and (or) between groups, and are applicable to an imide-ized reaction in the form of gas, a solution, for example, a water solution, and others. Generation imide is N-permutation imide when an imide-ized agent is an amine.

[0012] Although these imide-ized agents can transform a maleic anhydride to the imide of correspondence under the given heating-pressurization-shearing force impression conditions, they can also use an imide-ized catalyst if needed. Although each compound which can promote the reaction which changes an acid anhydride into the imide of correspondence as an imide-ized catalyst is usable, a typical group of such an imide-ized catalyst is tertiary amine. As tertiary amine useful as an imide-ized catalyst, there are a trimethylamine, triethylamine, tripropylamine, N,N-dimethylaniline, a pyridine, etc.

[0013] Imide-ization by <imide-ized (the 1)> this invention is performed under the conditions of heating-pressurization-shearing force impression. And after this process (it is called the first process), an imide-ized reactant is given to reduced pressure conditions, and the process (the second process) which removes a low-

molecular contaminant, for example, the used imide-ized agent, the used solvent or a dispersion medium, a by-product, and others is carried out. The pressure at the time of imide-izing an imide-ized precursor copolymer at the first process in this invention is 10 kg/cm². It holds above. In order to hold a pressure in a predetermined value thru/or the predetermined range, it is 10 kg/cm² to the kneading band of an extruder. A screw element is constituted so that the above pressure may be added, and it is the brewing pressure of the imide-ized agent to this kneading band 10 kg/cm². What is necessary is just to maintain above. A pressure is 10 kg/cm². It is because an imide-ized agent evaporates in a kneading field, and it does not enter in the fused precursor copolymer, kneading effectiveness falls, as a result desired imide-ized effectiveness is not attained in the following.

[0014] The temperature at the time of imide-izing a raw material copolymer at the first process in this invention is chosen from the 200-degree-C or more range of 270 degrees C or less. It is because the fall of the molecular weight of a precursor copolymer becomes remarkable, and the ratio of reverse reaction will increase since the imide-ized reaction is reversible and imide-ized effectiveness will fall on the contrary, if reaction temperature exceeds 270 degrees C. It is because kneading effectiveness with an imide-ized agent will fall for hyperviscosity, as a result desired imide-ized effectiveness will not be attained, even if heating melting of a precursor copolymer becomes being temperature lower than 200 degrees C inadequate on the other hand or it fuses.

[0015] The kneading conditions at the time of imide-izing a precursor copolymer at the first process in this invention are chosen from the shear rate range not more than more than 10 sec-1 to 1250 sec-1. Since both uniform kneading is not made when the shear rate given carries out melting kneading of the raw material copolymer with an imide-ized agent in the condition of not amounting to 10 sec-1, containing a copolymer part with the low rate of imide-izing in part is not avoided without obtaining the imide-ized copolymer imide-ized by homogeneity. In order that the part into which this imide-ization is not performed enough may naturally receive the fall of the molecular weight by the heat history at the time of melting kneading, the imide-ized copolymer obtained cannot have uniform high molecular weight, becomes in a part, and will contain the copolymer part of low molecular weight. Although this rate of the reduction in imide / low-molecular-weight copolymer contained in part seldom affect high temperature oxidation stability-proof, since it has big effect to the impact strength-proof of blend resin with ABS plastics etc., it is difficult for the imide-ized copolymer imide-ized under low shear rate conditions to give blend resin with the ABS plastics which have high shock resistance. In the case of the shear rate which surpasses 250 sec-1 on the other hand, since the molecular weight fall of the copolymer for local shear heating is caused shortly, it is not desirable too. In addition, it is the especially desirable property of the imide-ized copolymer by this invention to have uniform high molecular weight and an imide content, and it is understood as the ability to have not attained by the conventional approach. That is, the imide-ized copolymer obtained by this invention has the molecular weight with most almost same copolymer molecules, and an imide content, consequently, in addition to high thermal resistance or high temperature oxidation stability-proof, can have blend resin of it, ABS plastics, etc. in accordance

with high shock resistance. Moreover, as compared with the conventional approach near and this cause the fall of molecular weight extremely to the molecular weight of the maleic-anhydride copolymer whose molecular weight of the imide-ized copolymer by this invention is the precursor, it is an extraordinary advantage similarly.

[0016] It is limited within 5 minutes 2 minutes or more, another reaction condition, i.e., the heating mixing time, of imide-izing by this invention. It is because productive efficiency will also fall, kneading effectiveness will fall under for 2 minutes on the other hand and high imide-ized effectiveness will not be attained at an elevated temperature 200 degrees C or more, while the molecular weight of a copolymer falls if for 5 minutes is exceeded. As for the amount of the imide-ized agent used, it is desirable that it is 0.8-1.5 mols per one mol of maleic anhydrides in a copolymer. moreover, since degradation of a generation imide-ized copolymer, coloring of the precursor copolymer, or a molecular weight fall is prevented at imide-ized reaction time or the time of kneading (detail special mention) in the **** insertion band of carrying out as the preceding paragraph process, a phenol system stabilizer, the Lynn system stabilizer, or (and) a sulfur system stabilizer can be used together as an antioxidant.

[0017] By imide-ization by this invention, after imide-izing a precursor copolymer at the first process, a low-molecular contaminant is removed from an imide-ized copolymer at the second process. The approach of removal of a low-molecular contaminant can be enforced using the usual technique according to the class of the by-product contained in the imide-ized copolymer, unreacted object, etc. For example, by the **** vent method, the imide-ized copolymer is made into a melting condition, and reduced pressure (a vacuum is included) is applied to this, and it can heat further to coincidence and can carry out to it. When applying a vacuum, a degree of vacuum can be set to 50 - 200Torr.

[0018] <Imide-ized (that 2)> drawing 1 shows roughly the equipment for enforcing the continuation manufacturing method of an imide-ized copolymer based on this invention. This equipment 1 is the said direction rotation completeness engagement mold twin screw extruder, and has the feed hopper 5 and the preliminary mixing region 6 of an imide-ized precursor copolymer. In the kneading band 3 It has the pump 7 which presses an imide-ized agent fit, and the imide-ized agent feed hopper 8 linked to this pump 7. Further between the insertion band 2 and the kneading band 3 and between the kneading band 3 and the removal band 4 The seal elements 13 and 14 for maintaining a desired imide-ized pressure are arranged. Vent-ports 9 and 10 are formed in the removal band 4, and the vent-port is connected to the vacuum pump 12 through the trap 11. The nozzle opening (die) 15 is formed in the outlet of the removal band 4.

[0019] As for imide-izing performed using such equipment, it is common to carry out by the approach of consisting of inserting an imide-ized precursor copolymer in the insertion band which is open for free passage to a kneading band continuously or intermittently, feeding a kneading band with this continuously or intermittently from this insertion band, and feeding a removal band with the imide-ized resultant acquired by kneading with an imide-ized agent and doing in this way continuously or intermittently. This invention can be carried out using various equipments in

addition to the above-mentioned example of continuous system equipment. For example, there are continuous system equipment and various batch type equipments, such as a kneader various kinds of extruders, such as a single screw extruder, an engagement mold twin screw extruder, and a non-engagement mold twin screw extruder, a deformation screw, and barrel type or a non-screw type ram extruder. As equipment in which desirable operation is possible, there is a said direction rotation completeness engagement mold twin screw extruder. The kneading effectiveness is high, and this does not have stagnation (self-cleaning nature is), and uniform kneading is possible for it, and it is because it generates thru/or is moreover easy to maintain a pressure to a kneading field. Moreover, imide-ization by this invention can also be carried out with the equipment which omitted the insertion band in the equipment of drawing 1. That is, an imide-ized precursor copolymer is supplied to the kneading band 3 with an imide-ized agent separately from an imide-ized agent.

[0020]

[Example] The following examples and examples of a comparison are for explaining this invention still more concretely. This invention is not limited to the following examples, unless that summary is exceeded.

[0021] They are 2.0kg / HR about the mixed liquor to which example 1 content volume added the G tert-butylperoxy acetate 0.011 weight section to the stirring reactor of the complete-mixing mold which is 2l. to a total of 100 weight sections of 88.9 % of the weight of styrene, 4.0 % of the weight of methyl methacrylates, and 7.1 % of the weight of methyl ethyl ketones. It supplied continuously at the rate. It is the maleic-anhydride monomer heated to the same reactor as coincidence at about 70 degrees C 0.4 kg/HR It supplied continuously at the rate. Polymerization temperature was made into 150 degrees C, the polymerization was advanced to 65 % of the weight of polymerization invert ratios, and polymerization reaction mixture was taken out continuously.

[0022] The polymerization reaction mixture taken out continuously was immediately sent to the devolatilizer of 200 degrees C / 50Torr, and the unreacted monomer and the solvent were separated and pelletized. As a result of analysis, the presentation of styrene / maleic anhydride / methyl methacrylate was weight %, and the obtained pellet was the ternary polymerization object of 69/26/5. It is imide-ized precursor copolymerization-ization which is the ternary polymerization object acquired by this as above-mentioned using the 30mm said direction rotation completeness engagement mold twin screw extruder as shown in drawing 1 5 kg/HR It supplied at the rate. Most consists of knee DINGU disks and, as for the kneading band of this extruder, those both ends are equipped with the seal element. Imide-ized extrusion conditions were set up so that temperature might become [the mean flow-time of 50 sec-1 and a kneading band] 250 degrees C and a shear rate might become in about 3 minutes.

[0023] The shear rate (S) here was determined based on the degree type.

$S = \pi DN/h$ D:screw-diameter N:screw-speed h: The mean flow-time of a screw channel depth and a kneading band was measured by the approach as shown below. Extruding a raw material copolymer under some extrusion conditions, black or a red coloring agent masterbatch is added from the feed hopper 5 of a copolymer pellet,

and change of the color of the melting polymer in the imide-ized agent feed hopper 8 and the first vent-port 9 which have been changed into both open conditions is observed. Mean flow-time RT is calculated for the time amount in each part which it begins to color, and the time amount whose coloring by the coloring agent is lost completely by the degree type as T (8-**), T (8-**), T (9-**), and T (9-**).

$$RT = \{(T(9-**) - T(8-**)) + (T(9-**) - T(8-**))\} / 2$$

[0024] An aniline is used as an imide-ized agent and it is this from the feed hopper 1.2 kg/HR. The kneading band was supplied at the rate. the discharge pressure of the aniline brewing pump at this time -- 12 kg/cm² it was. Reduced pressure of about 100 torr(s) was applied to both vent-ports, and separation recovery of sub**** and the unreacted aniline from the vent-port was carried out at the trap. From the nozzle, melting resin was made into the shape of a strand, was extruded, cooled this strand in the tank, cut it by the cutter, and was made into the pellet type. The obtained imide-ized copolymer had 90% of imide-ized yield, as a result of carrying out quantitative analysis with an infrared spectrophotometer. The physical properties of the obtained imide-ized copolymer were as being shown in the 1st table. It is the mixture of the weight ratios 1/1 of the imide-ized copolymer and high shock resistance ABS plastics (Monsanto formation "tough REXX 710" (acrylonitrile: 24%, butadiene: 21%, styrene: 55%, and MFR: 8g / 10 minutes (240 degree-C/10kgf), VICAT: 90 degree C) by incorporated company) which were obtained 30 m/m for kneading. Compounding was carried out using the said direction rotation completeness engagement mold twin screw extruder, and the heat-resistant-resin constituent was obtained. The physical properties of the above-mentioned resin constituent were as being shown in the 2nd table.

[0025] In this example, the imide-ized copolymer and the heat-resistant-resin constituent were similarly manufactured except having changed temperature into 270 degrees C among imide-ized extrusion conditions, and having changed the shear rate into example 2 example 1 in the example of a publication, 30 sec-1. The obtained imide-ized copolymer had 80% of imide-ized yield. The physical properties of the obtained imide-ized copolymer are shown in the 1st table, and the physical properties of a heat-resistant-resin constituent are shown in the 2nd table.

[0026] In the example of a publication, a shear rate is changed into example 3 example 1 15 sec-1 among imide-ized extrusion conditions. Moreover, the outside which used N of the 0.5 weight section (they are criteria about the maleic-anhydride residue in a precursor copolymer) and N-dimethyl dodecyl amine (DMDA) other than an aniline (they are tales doses when it can set for this example) as an imide-ized catalyst as an imide-ized agent. In this example, the imide-ized copolymer and the heat-resistant-resin constituent were manufactured similarly. The obtained imide-ized copolymer had 95% of imide-ized yield. The physical properties of the obtained imide-ized copolymer and a heat-resistant resin constituent were as being shown in the 1st table and the 2nd table.

[0027] The outside which changed the shear rate into example of comparison 1 example 1 among the imide-ized extrusion conditions of a raw material copolymer in the example of a publication 8 sec-1 manufactured the imide-ized copolymer and the heat-resistant-resin constituent similarly in this example. The obtained imide-ized copolymer had 88% of imide-ized yield. The physical properties of an imide-

ized copolymer are shown in the 1st table, and the physical properties of a heat-resistant-resin constituent are shown in the 2nd table.

[0028] The outside which changed the shear rate into example of comparison 2 example 2 among imide-ized extrusion conditions in the example of a publication 8 sec-1 manufactured the imide-ized copolymer and the heat-resistant-resin constituent similarly in this example. The obtained imide-ized copolymer had 81% of imide-ized yield. The physical properties of an imide-ized copolymer are shown in the 1st table, and the physical properties of a heat-resistant-resin constituent are shown in the 2nd table.

[0029] The outside which changed the shear rate into example of comparison 3 example 3 among imide-ized extrusion conditions in the example of a publication 8 sec-1 manufactured the imide-ized copolymer and the heat-resistant-resin constituent similarly in this example. The obtained imide-ized copolymer had 92% of imide-ized yield. The physical properties of an imide-ized copolymer are shown in the 1st table, and the physical properties of a heat-resistant-resin constituent are shown in the 2nd table.

[0030]

[Table 1]

第1表

| | 原料(1) (S/MA/MMA) | 実 施 例 | | | 実 施 例 | | |
|-------------------------------|---------------------|--------------------|------|----------------------------|-------|------|-------------|
| | | 1 | 2 | 3 | 1 | 2 | 3 |
| 混練温度 ℃ | | 250 | 270 | 250 | 250 | 270 | 250 |
| 混練時間 分・秒 | | 2.40 | 3.05 | 3.30 | 3.10 | 3.20 | 3.15 |
| 剪断速度 sec ⁻¹ | | 50 | 30 | 15 | 8 | 8 | 8 |
| 使用イミド化剤 | | ALN ^{註2)} | ALN | ALN | ALN | ALN | ALN |
| イミド化剤圧力 kg/cm ² | | 12 | 12 | 12 | 12 | 12 | 12 |
| イミド化剤モル比 ^{註1)} | | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| 使用触媒(量) (部) | | 無 | 無 | DMDA ^{註3)} 0.5 | 無 | 無 | DMDA 0.5 |
| イミド化収率 ^{註1)} モル% | 0 | 90 | 80 | 95 | 88 | 81 | 92 |
| 分子量(×10 ⁴) | 13.5 | 13.3 | 13.0 | 13.1 | 12.5 | 12.5 | 12.7 |
| ビカッ卜軟化点 ℃ | 145 | 156 | 155 | 157 | 155 | 155 | 156 |

註1) MA (無水マレイン酸) を基準として計算

註2) アニリン

註3) N, N-ジメチルドデシルアミン

[0031]

[Table 2]

第2表

| | 実 施 例 | | | 実 施 例 | | |
|---|-------|-----|-----|-------|-----|-----|
| | 1 | 2 | 3 | 1 | 2 | 3 |
| 引張り強さ (Kg/cm ²) | 485 | 480 | 485 | 480 | 480 | 485 |
| 引 張 り 伸 び (%) | 35 | 30 | 35 | 20 | 25 | 25 |
| 曲げ強さ (Kg/cm ²) | 845 | 840 | 845 | 840 | 845 | 845 |
| 曲げモジュラス (10 ⁴ Kg/cm ²) | 2.8 | 2.8 | 2.8 | 2.8 | 2.8 | 2.8 |
| IZOD衝撃強さ (Kg-cm/cm) | 7.5 | 7.0 | 7.5 | 4.5 | 4.5 | 5.0 |
| F D I (Kg-cm) | 50 | 45 | 50 | 15 | 15 | 20 |
| H D T U L (℃) | 105 | 105 | 105 | 105 | 105 | 106 |
| V I C A T (℃) | 121 | 120 | 121 | 120 | 120 | 121 |
| MFR (g/10分) 註1) | 20 | 21 | 20 | 21 | 21 | 21 |

[0032] As shown in the 1st table, the imide-ized copolymer obtained according to the manufacturing method of this invention and the imide-ized copolymer obtained by the example of a comparison are hardly (imide-ized yield, molecular weight, thermal resistance) accepted for a difference in the physical properties as an imide-ized copolymer. However, as shown in the 2nd table, the blend resin constituent of the imide-ized copolymer and ABS plastics which were obtained according to the manufacturing method of this invention shows the outstanding physical-properties balance, and the blend resin constituent of the imide-ized copolymer and ABS plastics which were obtained from the example of a comparison is greatly inferior in shock resistance to having high shock resistance so that especially IZOD impact strength and FDI may see.

[0033] In addition, measurement of the physical properties indicated to the 1st table and the 2nd table was performed based on the following approaches.

1) Molecular weight (weighted mean) : gel permeation chromatography 2
 VICAT(BIKATTO softening temperature):JIS K-72063 It is :JIS in tension
 strength: K-71134 Tension elongation: JIS K-71135 bending strength: JIS K-72036
 bending modulus: JIS K-72037 IZOD impact strength: JIS K-71108 FDI(falling
 weight impact strength):JIS K-72119 HDTUL(heat-deflection-
 temperature/deflection temperature under load):JIS K-720210 MFR(melt flow
 rate):JIS K-7210 [0034]

[Effect of the Invention] It is as having described above in the term of "the outline of invention" that the imide-ized copolymer of the amount of macromolecules which has thermal resistance and high temperature oxidation stability-proof can

manufacture economically continuously by one paragraph of a non-drainage system according to this invention.

[Translation done.]